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GB 1067241 A GB 0922648 A EP 0350252 A1

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(54) Chromatography column

(57) A chromatography column assembly comprises a pair of transparent glass plates (1, 2) positioned so that a major surface of each (4, 5) faces, is parallel to, and is spaced from the other. An adhesive (3) is provided between the plates to secure each to the other. This adhesive is patterned by screen printing to define a channel (6) between, and running parallel to, the major surfaces of the plates. The channel has an inlet and an outlet orifice drilled through the plates for the introduction and exhaust of a sample fluid. The channel may be packed with an inorganic filler to increase column capacity. The channel need not be straight, but as in Fig. 3 (not shown), may double back on itself several times.

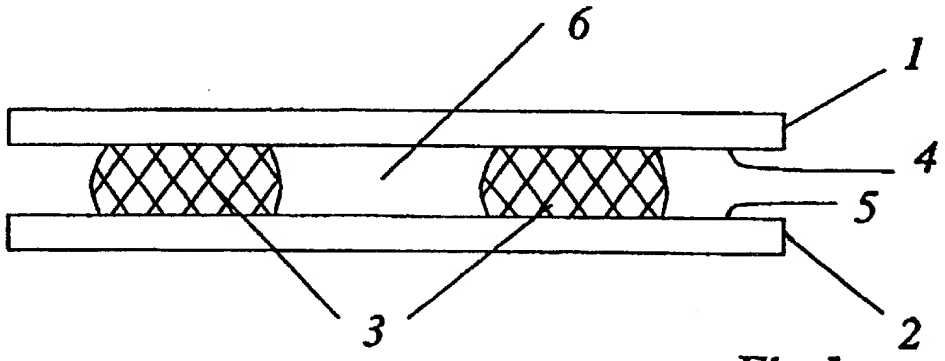

Fig.1.

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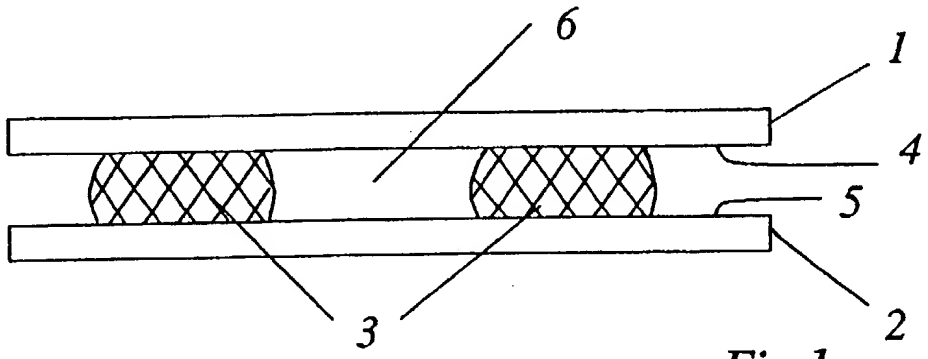

Fig.1.

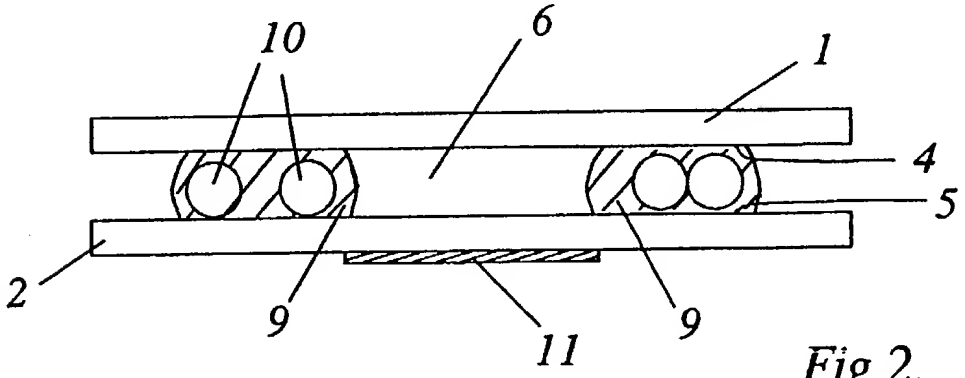

Fig.2.

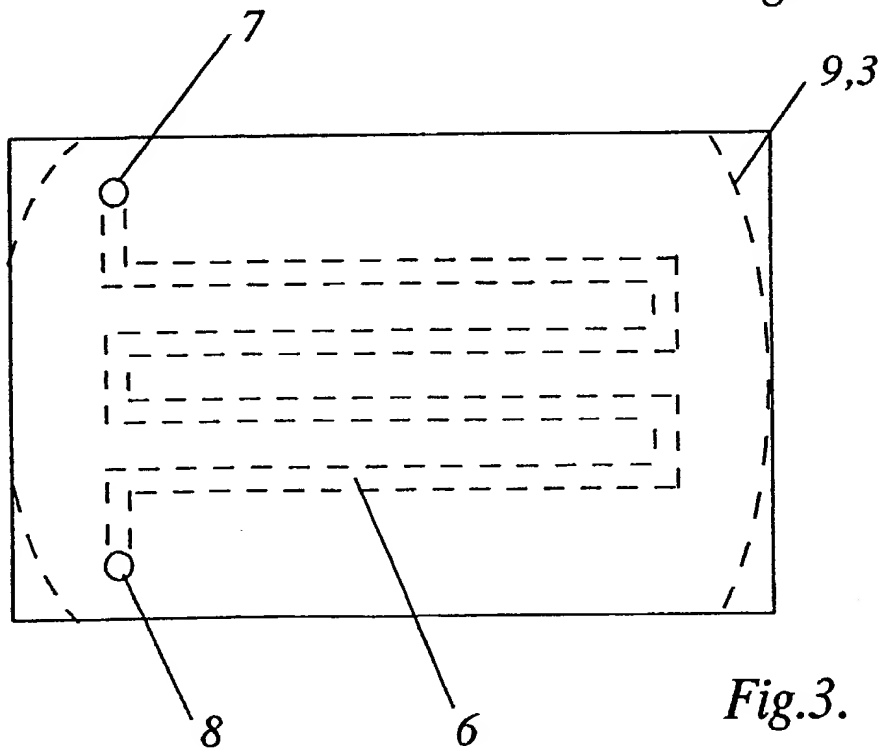

Fig.3.

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CHROMATOGRAPHY COLUMN ASSEMBLY

This invention relates to fluid conduit assemblies, and particularly to fluid conduit assemblies for use as columns in liquid or gas chromatography.

US Patent Number 4,474,889 discloses fluid conduit assemblies made from a silicon wafer which has had grooves etched into one surface, and which has a plate with a substantially planar surface placed in intimate contact with or glued to the grooved wafer to define a channel. This channel is used as the column of a liquid or gas chromatographic system in place of the long narrow bore capillary tubes usually employed.

The assemblies described above have a number of disadvantages. For example, the silicon wafer is fragile and expensive, the cross sectional area of the channel may vary due to its method of manufacture or the presence of different thicknesses of glue, and manufacturing costs are relatively high.

An object of the present invention is to enable the above disadvantages to be mitigated.

According to the invention there is provided a chromatography column assembly comprising a pair of plates relatively positioned so that a substantially flat major surface of each plate (a) faces, (b) is substantially parallel to, and (c) is spaced from, a substantially flat major surface of the other, and material between the said major surfaces securing each

to the other, the material being positioned to define a fluid conduit between and running parallel to the said major surfaces, the fluid conduit being provided with first and second orifices for the introduction and exhaust of a fluid respectively.

Such a chromatography column assembly can be easier and cheaper to manufacture than the column disclosed in US 4,474,889 and can provide better dimensional control.

Embodiments of the invention will now be described, by way of example only, with reference to the accompanying diagrammatic drawings in which:-

Figure 1 shows a schematic cross section of a first embodiment of the invention;

Figure 2 shows schematic cross section of a second embodiment of the invention; and

Figure 3 is a plan view of the embodiments of Figs 1 and 2.

A chromatograph column assembly comprises a pair of plates 1,2 which are relatively positioned so that a substantially flat major surface 4,5 of each plate (a) faces, (b) is substantially parallel to, and (c) is spaced from a substantially flat major surface 5,4 of the other. Material 3 provided between the said major surfaces 4,5 secures each to the other, and is moreover positioned to define a fluid conduit 6 between and running parallel to the said major surfaces 4,5. The fluid conduit is provided with an inlet orifice 7 (shown in Figure 3) and an outlet orifice 8 (also shown in Figure 3) for the introduction and exhaust of a fluid respectively.

In this first embodiment the plates 1 and 2 are made of

transparent glass and are each 7cm x 10cm x 2mm thick. The material 3 is an anaerobically curing epoxy resin which, during manufacture, is deposited onto the surface of one plate as a pattern by screen printing through a stencil. The second plate is then placed on top and the resin is left to cure. Before assembly the orifices 7,8 are drilled through one plate. The cross sectional dimensions of the conduit after assembly are approximately 100μm x 100μm. The final dimensions of the conduit may differ from the deposited dimensions if the resin creeps before or during cure. This factor must be taken into account if the final dimensions are important.

In the second embodiment shown in Figure 2 the assembly is again formed using a pair of transparent glass plates, but now the material used is a screen-printable mixture of a UV curing adhesive 9 (Norland Optical Associates UV sealant number 91) with spherical resin spacers 10. The spheres used in this case were EPOSTAR GP90 spacers, supplied by Nippon Shokubai Kagaku Kogyo Co Ltd, having diameters of approximately 9 microns.

During manufacture the mixture of spheres and UV curing adhesive is deposited onto the substantially flat major surface of one plate by screen printing through a suitable stencil. The second plate is then placed on top of the adhesive and clamped in place. The adhesive and spherical spacers mixture is thus put under pressure, and the adhesive spreads until the separation of the plates is only slightly larger than the diameter of the spheres. The adhesive is then cured by exposure to ultra violet light.

Inlet and outlet orifices which communicate with the conduit

so formed are drilled as before. In this embodiment nozzles (not shown) are fixed firmly into the orifices to facilitate the attachment of a fluid supply. Of course the orifices may, as an alternative, be formed after assembly. In addition, a thin film heating element 11 has previously been applied to one side of one of the glass plates to enable parts of the assembly to be heated to facilitate desorption of gas molecules from the conduit walls.

One of many possible alternatives for the material 9 of Fig. 2 together with the spacers 10 is a screen-printable ink formed by thorough mixing of 2 parts by weight H102/1/grade 15 soda glass spherical spacers (supplied by Jencons Scientific Ltd, Leighton Buzzard, England) having diameters of the order of 0.10mm, with 98 parts by weight Frit S261 (a finely powdered lead borate glass supplied by Blythe Colours, Johnson Matthey, Cresswell, Stoke on Trent, England) and sufficient medium 63/182 (also obtainable from Blythe Colours) to form a smooth paste suitable for screen printing. This ink may be screen printed onto a soda lime glass plate in the required pattern, and then pre-fired by heating in air with a temperature ramp of 20°C per minute from room temperature to 400°C, followed by holding the temperature steady at 400°C for 10 minutes, and then cooling to room temperature with a temperature ramp of 10°C per minute. This heat treatment causes any organic binder present to be burnt off, and melts the frit so that it adheres to the plate onto which it has been printed. The soda-lime glass spacers do not melt or deform significantly during this treatment as they have a higher melting point and glass transition temperature than the melting point of the frit.

The plate with the pre-fired pattern is then placed on top of a second clean soda lime glass plate so that the printed pattern lies between the two plates. This assembly is then fired again to fuse the plates and the pre-fired pattern, forming a fluid conduit. The conditions for this 'assembly' fire are an initial temperature ramp up from room temperature to 400°C at 20°C per minute, followed by holding the temperature steady at 400°C for 10 minutes, and then cooling back to room temperature with a temperature ramp of 10°C per minute.

Using this last technique, it is possible to manufacture conduits which are filled with inorganic packing material. This may have the advantage of increasing the surface area within the conduit, thus increasing the column capacity. A packing material sensitive to a specific chemical species of interest may also be employed to enhance the detection of trace quantities. This filling may be accomplished by depositing a powdered inorganic material on top of the plate with the pre-fired glass pattern and spacers. Excess material may then be removed using a doctor blade. A second clean soda lime glass plate is then placed on top of the first plate. The assembly is subsequently clamped and re-fired as described above.

Although in the above embodiments the communicating orifices are formed by drilling through one of the plates, they may, as an alternative, be formed by bringing the conduit up to the edge of the plates to allow the fluid to be injected or exhausted in the plane of the conduit. Once again nozzles may be affixed to the orifices to make the connection of fluid lines easier.

If desired, the conduits may be configured to have different

cross-sectional areas in different regions, thereby forming reservoirs (filled with packing material if required) to enable the column to store specific chemical species which may be released by heating the whole (or a portion of) the assembly. Thus the assembly may be used as a sampling system in which the concentration of certain chemical species can be increased to facilitate detection.

The packing material described in the second embodiment above may, as an alternative, be deposited by screen printing and firing or by other methods of selective deposition or deposition and selective removal.

Although spherical spacers are used in the embodiments described with reference to Figs. 2 and 3, other spacing means may alternatively be employed, for example rods, shims or polyhedra made of metal or ceramics other material. Moreover, a very wide range of materials may be substituted for the specific adhesive materials described in the examples, and the material may be positioned in a pattern using a method of selective deposition or deposition and selective removal. Furthermore, the plates used need not be made of glass; plastics, metals and other materials can be used with little modification of the techniques of manufacture described. Such column assemblies may be used in gas or liquid chromatography.

CLAIMS

1. A chromatography column assembly comprising a pair of plates relatively positioned so that a substantially flat major surface of each plate (a) faces, (b) is substantially parallel to, and (c) is spaced from, a substantially flat major surface of the other, and material between the said major surfaces securing each to the other, the material being positioned to define a fluid conduit between and running parallel to the said major surfaces, the fluid conduit being provided with first and second orifices for introduction and exhaust of a fluid respectively.

2. A chromatography column assembly as claimed in claim 1 in which the material incorporates spacers for the two plates.

3. A chromatography column assembly as claimed in claim 1 or claim 2 in which the fluid conduit contains packing material.

4. A chromatography column assembly as claimed in claim 1 or claim 2 or claim 3 in which the fluid conduit cross sectional area varies along its length.

5. A chromatography column assembly as claimed in any one of claims 1 to 4 in which a heater element is provided on a portion of a surface of a said plate.

6. A chromatography column assembly substantially as herein described with reference to Figures 1 and 3, or 2 and 3 of the drawings.

Patents Act 1977
Examiner's report to the Comptroller under Section 17 (The Search Report)

Application number: GB 9303718.2

Relevant Technical fields

(i) UK Cl (Edition L) B1H

(ii) Int Cl (Edition 5) B01D, G01N

Search Examiner

J H WARREN

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASE: WPI

Date of Search

23 MARCH 1993

Documents considered relevant following a search in respect of claims 1-6

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
Y	GB 1067241 A (INTERLECTRON) Page 3, lines 62-72	1, 3, 4
Y	GB 0922648 A (BAIRD AND TATLOCK) Figures 5, 6, lines 36-59 page 2	1, 2, 3, 5
Y	EP 0350252 A1 (KODAK) Claim 1	1, 2

Category	Identity of document and relevant passages	Relevant to claim(s)

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

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